SHORT COMMUNICATION

IDENTIFICATION OF SCOPOLETIN IN HYDROLYSATES OF WEIGELA (CAPRIFOLIACEAE)

C. W. GLENNIE and B. A. BOHM

Department of Botany, University of British Columbia, Vancouver 8, Canada

(Received 6 December 1967)

Abstract—Scopoletin (6-methoxy-7-hydroxycoumarin) has been identified as a major phenolic constituent in hydrolysates of two species and one hybrid of *Weigela* (Caprifoliaceae).

REPORTS of the occurrence of coumarins in members of the Caprifoliaceae are not common. Hegnauer¹ included only two such compounds in his review: fraxin from *Diervilla canadensis* Willd. and *D. diervilla* Moem. and esculetin from *Symphoricarpus occidentalis* Hook. and *S. rivularis* Suksdorf. Recently, Hörhammer and his co-workers² reported the presence of scopoletin in *Viburnum prunifolium* L. In a comparative biochemical study of some members of the Caprifoliaceae, Plouvier³ observed a blue fluorescence on chromatograms of extracts of *Weigela cornaenais* Thunb. which was attributed to esculetin although no structural studies of the compound were reported. In our laboratory, a bright blue u.v.-fluorescent spot was observed on chromatograms of hydrolysed extracts of *W. floribunda* C. A. Mey, *W. florida* (Bunge) DC., and *W.* "Bristol Ruby" × "Abel Carriere". The compound responsible for the fluorescence was isolated and identified as scopoletin.

EXPERIMENTAL

Leaf material, collected from plants growing on the campus, was exhaustively extracted with boiling 80 per cent ethanol and the combined extracts were evaporated to dryness. The residue was extracted with hot water, and the extract was filtered through Celite, made 2 N with conc. HCl and hydrolysed by heating for 30 min. The hydrolysate was continuously extracted with ether for 12 hr. Evaporation of the ether left a syrup which was taken up in a small vol. of ethanol and applied as spots to sheets of Whatman 3 MM paper and subjected to two-dimensional chromatography, using the organic layer of benzene: acetic acid: water (10:7:3) and then 2 per cent formic acid in water. The spots of R_f s 0·38/0·34, located by their fluorescence, were cut out and eluted with ether. After removal of the ether the residue was sublimed at $100^{\circ}/0·15$ mm. The sublimate was recrystallized from methanol and water to yield colourless crystals, with a small amount of brown residue removed by brief washing with cold ether. Isolated material was identical in m.p., R_f , u.v. and i.r. spectra with authentic scopoletin. Melting points were: isolated $201-202^{\circ}$, authentic $201\cdot5-3\cdot0^{\circ}$, mixed $200-201^{\circ}$ and lit.⁴ 204° .

Acknowledgement—We wish to thank the National Research Council of Canada for financial support.

¹ R. HEGNAUER, Chemotaxonomie der Pflanzen, Bd. III, p. 367. Birkhäuser Verlag, Basel und Stuttgart (1964).

² L. HÖRHAMMER, H. WAGNER and H. REINHARDT, Z. Naturforsch. 22b, 768 (1967).

³ V. PLOUVIER, Compt. Rend. 237, 1013 (1951).

⁴ Handbook of Chemistry and Physics, 47th edition, p. C-261. Chemical Rubber Co., Cleveland (1966-67).